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भारतीय मानक

रोगन के लिए जिंकक्रोम — विशिष्टि

(चौथा पुनरीक्षण)

Indian Standard

ZINC CHROME FOR PAINTS — SPECIFICATION

(Fourth Revision)

ICS 71.100; 87.060

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BUREAU OF INDIAN STANDARDS MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG NEW DELHI 110002 Raw Materials for Paint Industry Sectional Committee, CHD 021

FOREWORD

This Indian Standard (Fourth Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Raw Materials for Paint Industry Sectional Committee had been approved by the Chemical Division Council.

This standard was first issued in 1950 and was subsequently revised in 1963, 1966 and 1979. In this revision Type 1 zinc chrome has been deleted, not being in vogue and the other types redesignated.

The composition of the committee responsible for the formulation of this standard is given in Annex B.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2: 1960 'Rules for rounding off numerical values (revised)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

ZINC CHROME FOR PAINTS — SPECIFICATION

(Fourth Revision)

1 SCOPE

This standard prescribes requirements and methods of sampling and test for zinc chrome for paint industry.

2 REFERENCES

The Indian Standards listed below contain provisions which through reference in this text, constitute provisions of this Indian Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision and parties to agreements based on this Indian Standard are encouraged to investigate the possibility of applying the most recent editions of the Indian Standards indicated below:

IS No.	Title
33 : 1992	Methods of sampling and testing for inorganic pigments and extenders for paints (third revision)
264 : 1976	Nitric acid (second revision)
266 : 1993	Sulphuric acid (third revision)
323:1959	Rectified spirit (revised)
1070 : 1992	Reagent grade water (third revision)
1303 ; 1983	Glossary of terms relating to paints (second revision)
2316 : 1990	Methods of preparation of standard solutions for colorimetric and volumetric analysis (second revision)

3 TERMINOLOGY

For the purpose of this standard, the definitions given in IS 1303 and 3 of IS 33 shall apply.

4 TYPES

4.1 The material shall be of the following two types:
Type 1 — Basic potassium zinc chromate of approximate composition K₂CrO₄, 33 ZnCrO₄, Zn(OH)₂, intended for use mainly as anti-corrosive pigment for its rust-inhibiting properties in general.

Type 2 — Basic zinc chromate free from alkali metals of approximate composition ZnCrO₄, 4Zn(OH)₂, intended for use mainly as anti-corrosive pigment for its rust-inhibiting properties generally in etch primers.

5 REQUIREMENTS FOR ZINC CHROME

5.1 Form and Condition

The material shall be in the form of dry powder or in such condition that it is possible to reduce it to powder form by crushing, without grinding action, under a pallette knife.

5.2 Composition

5.2.1 Type 1

The material shall consist essentially of basic potassium zinc chromate and shall be of approximate composition K₂CrO₄, 3ZnCrO₄, Zn(OH)₂.

5.2.2 Type 2

The material shall consist essentially of basic zinc chromate free from alkali metals and shall be of approximate composition ZnCrO₄, 4Zn(OH)2.

- **5.2.3** The material shall be free from organic dyestuff or substitute of any kind and shall be completely soluble in 10 percent acetic acid at a temperature of 80° C
- **5.4** The material shall also comply with the requirements given in Table 1.

5.5 Lead-Free Material

When lead-free material is required, the material shall also be tested for freedom from lead in accordance with the procedure prescribed in 26 of IS 33. When thus tested, the material shall not contain lead or lead compounds or a mixture of both, calculated as metallic lead (Pb), exceeding 0.03 percent.

6 PACKING AND MARKING

6.1 Packing

The material shall be suitably packed as agreed to between the purchaser and the supplier.

Table 1 Requirements for Zinc Chrome

(Clause 5.4)

SI	***************************************	Requirement		Method of Test, Ref to	
		Type 1	Type 2	Annex A of this Indian Standard	Cl of IS 33
(1)	(2)	(3)	(4)	(5)	(6)
i)	Volatile matter, percent by mass, Max	1.0	1.0		8
ii)	Residue on sieve, percent by mass, Max	0.5	0.5	_	9
iii)	Oil absorption, Min	15	15	_	10
iv)	Colour	Close match to the a sample	ipproved		11
v)	Matter insoluble in ammoniacal ammonium chloride solution, percent by mass, Max	0.5	0.5	A-2	
vi)	Combined water, percent by mass, Max	8.0		A-3	
vii)	Chromic anhydride (CrO ₃), percent by mass, Min	43.0	17	A-4	_
viii)	Total zinc content (as ZnO), percent by mass	36.5 to 40	68.5 to 72	.0 A-4	_
ix)	Alkali metal content (as K2O), percent by mass	10.0 to 12.0		A-5	
x)	Water soluble sulphates (as SO ₄), percent by mass, Max	0.1	0.1	A-6	_
xi)	Water soluble chlorides (as Cl), percent by mass, Max	0.1	0.1	A-6	_
xii)	Water soluble nitrates (as NO ₃), percent by mass, Max	0.1	0.1	A-6	
xiii)	Water soluble chromates (as CrO ₃), percent by mass	0.25 to 0.50	_	A-7	_
xiy)	Matter soluble in water, percent by mass, Max		0.5	_	19

6.2 Marking

The containers shall be marked with the following information:

- a) Name and type of material;
- b) Indication of the source of manufacture;
- c) Net mass of the material;
- d) Batch number or lot number in code or otherwise; and
- e) Month and year of manufacture.
- **6.2.1** The containers shall also be marked 'LEAD FREE' as prescribed in **27.2.2** of IS 33, when the material is lead-free.

6.2.2 BIS Certification Marking

The product may also be marked with the Standard Mark.

6.2.2.1 The use of the Standard Mark is governed by the provisions of *Bureau of Indian Standards Act*, 1986 and the Rules and Regulations made thereunder. The details of conditions under which the licence for the use of Standard Mark may be granted to manufacturers

or producers may be obtained from the Bureau of Indian Standards.

7 SAMPLING

7.1 Representative samples of the material shall be drawn as prescribed in **5** of IS 33.

7.2 Criteria for Conformity

The material shall be taken as conforming to this specification if the composite sample satisfies all the requirements prescribed.

8 TEST METHODS

8.1 Tests shall be conducted as prescribed in IS 33 and in Annex A, according to the requirements of Table 1 and **5.4**.

8.2 Quality of Reagents

Unless specified otherwise, pure chemicals and distilled water (see IS 1070) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

ANNEX A

(Table 1 and Clause 8.1)

ANALYSIS OF ZINC CHROME FOR PAINTS

A-1 PREPARATION OF SAMPLE

Grind the pigment, if it is lumpy or not finely ground, to a fine powder. If the sample is large, mix thoroughly and take a representative portion.

A-2 DETERMINATION OF MATTER IN-SOLUBLE IN AMMONIACAL AMMONIUM CHLORIDE SOLUTION

A-2.1 Outline of the Method

Matter insoluble in ammoniacal ammonium chloride solution is determined by drying the residue obtained after shaking the material with ammoniacal ammonium chloride solution.

A-2.2 Apparatus

A-2.2.1 Sintered Glass Crucible (G No. 4) or Gooch Crucible

A-2.3 Reagent

A-2.3.1 Ammoniacal Ammonium Chloride Solution

Dissolve 30 g of ammonium chloride in a mixture of 500 ml of ammonium hydroxide of relative density 0.92 and 600 ml of water.

A-2.4 Procedure

Shake approximately 5 g of the sample, accurately weighed, with 100 ml of the reagent for 1 h at room temperature. Allow to stand for 30 min and decant the supernatant liquid through a tared sintered glass or Gooch crucible. To the residue add a further 100 ml of reagent, shake for 5 min and filter the whole suspension through the same crucible, any adherent residue being transferred to the crucible by means of a policeman and with the minimum additional reagent. Wash the sides of the crucible and the residue with two successive portions of 25 ml water, then dry the crucible and the residue to constant mass in an oven maintained at $100 \pm 2^{\circ}$ C.

A-2.5 Calculation

Matter insoluble in ammoniacal ammonium chloride solution,

percent by mass =
$$\frac{100 \, m}{M}$$

where

m = mass in g of residue, and

M =mass in g of the sample taken for the test.

A-3 DETERMINATION OF COMBINED WATER

A-3.0 Outline of the Method

Combined water is determined by direct combustion of material at 1 000°C and making correction for the volatile matter.

A-3.1 Procedure

Place about 2 g of the sample, accurately weighed, in a porcelain boat. Introduce the boat into a combustion tube of an electrically heated combustion furnace of the type used for the determination of carbon by direct combustion. Connect the combustion furnace to a source of pure dry air at one end and an accurately weighed train of absorption tubes containing dry fused calcium chloride free from lime at the other end. Heat the combustion tube to 1 000°C and continuously pass a current of dry air. After heating for 1 h, cool the tube. Reweigh the absorption train. Repeat this operation till constant mass of the absorption train is obtained. The increase in mass gives the combined water and volatile matter present in the material.

A-3.2 Calculation

Combined water,

percent by mass =
$$\frac{m}{M} \times (100 - V)$$

where

m = increase of mass in g of the absorption train,

M = mass in g of the sample taken for the test,

V = percent by mass of volatile matter (see Table 1).

A-4 DETERMINATION OF CHROMIC ANHYDRIDE AND ZINC OXIDE CONTENT

A-4.0 Outline of the Method

Chromic anhydride is determined by titrating the iodine liberated from potassium iodide with standard sodium thiosulphate solution. Zinc oxide is determined in the same solution by titrating the iodine subsequently liberated on addition of potassium ferricyanide with standard sodium thiosulphate solution.

A-4.1 Reagents

A-4.1.1 Dilute Sulphuric Acid

Approximately 7 N.

A-4.1.2 Potassium Iodide Solution (Freshly Prepared)

10 percent.

A-4.1.3 Standard Sodium Thiosulphate Solution

0.1 N.

A-4.1.4 Starch Solution

0.5 percent.

A-4.1.5 Ammonia Solution

1:1(v/v).

A-4.1.6 Potassium Ferricyanide Solution

M/10.

Prepared by dissolving 8.231 3 g of potassium ferricyanide in 250 ml cold distilled water, filtered rapidly and kept in a dark bottle. The solution can be suitably used for 7 days if kept in dark when not in use

A-4.1.7 Ammonium Bifluoride (NH4 HF2)

Solid.

A-4.2 Procedure

- A-4.2.1 Weigh accurately about 0.5 g of the material in a 500-ml conical flask. Add 70 ml of water and 30 ml of dilute sulphuric acid. Boil slowly for 2 to 3 min and cool.
- A-4.2.2 Transfer the solution to a 500-ml iodine flask containing 100 ml of water and 20 ml dilute sulphuric acid. Add 30 ml of potassium iodide solution and allow to stand for 5 min. Titrate the liberated iodine with standard sodium thiosulphate solution after adding 5 ml of starch solution as indicator. Note the volume of sodium thiosulphate solution used in the titration (V_1) .
- A-4.2.3 After the titration for chromic anhydride, add ammonia solution dropwise until the litmus test paper first turns blue. (An excess of ammonia solution, more than 3 to 4 drops, should not be added as it is essential that the blue litmus should turn red with ammonium bifluoride to be added subsequently.) Cool and add 2 to 3 g of ammonium bifluoride and sufficient water to make up the volume to approximately 250 ml. (Sometime, traces of iodine liberated during neutralization can be cleared by adding one or two drops of sodium thiosulphate solution.) Add V1/3 ml of potassium ferricyanide solution. Stir and allow to stand for about 2 min. Titrate the liberated iodine with standard sodium thiosulphate solution. After the end point the colour of the test solution will be greenish yellow. Note the volume of sodium thiosulphate solution used in this second titration (V_2) .

A-4.2.3.1 In case V_2 is less than 6 ml, the titration is to be repeated with the addition of $V_2 + 2$ ml of potassium ferricyanide solution in place of $V_1/3$ ml.

A-4.2.3.2 In case V_2 is less than 3 ml, the test is to be repeated, starting from the sample, with the addition of 5 ml potassium ferricyanide solution.

A-4.3 Calculation

Chromic anhydride (CrO₃), percent by mass = $\frac{3.334 \times V_1 \times N}{M}$

Zinc content (as ZnO), percent by mass = $\frac{12.45 \times V_2 \times N}{M}$

where

 V_1 = volume in ml of sodium thiosulphate solution used in first titration,

 V_2 = volume in ml of sodium thiosulphate solution used in second titration,

N =normality of sodium thiosulphate solution used, and

M =mass in g of the material taken for titration.

A-5 DETERMINATION OF ALKALI METALS

A-5.0 Outline of the Method

Alkali metals are determined volumetrically by titrating the excess of standard ferrous ammonium sulphate in the alkali chromate solution, obtained by heating the material at high temperature, against standard potassium permanganate solution.

A-5.1 Reagents

A-5.1.1 Dilute Sulphuric Acid

Dilute 25 ml of sulphuric acid (see IS 266) by adding to water, cooling and making up to 100 ml with water.

A-5.1.2 Standard Ferrous Ammonium Sulphate Solution.

0.1 N (see IS 2316).

A-5.1.3 Standard Potassium Permanganate Solution, 0.1 N (see IS 2316).

A-5.2 Procedure

Heat 2.0 g of the material to approximately 600°C for 1 h. The material is thereby decomposed into zinc oxide, chromium sesquioxide (Cr₂O₃) and alkali metal chromate. Cool and extract the alkali metal chromate from the residue with hot water, filter and wash free from soluble chromate. Add a measured excess volume (usually 100 ml is sufficient) of ferrous ammonium sulphate solution and titrate the excess with standard potassium permanganate solution.

A-5.3 Calculation

Alkali metal (as K₂O), percent by mass

 $=0.1565(V_1-V_2)$

where

- V₁ is volume in ml of standard ferrous ammonium sulphate solution added, and
- V₂ is volume in ml of standard potassium permanganate solution required for titration.

A-6 DETERMINATION OF WATER-SOLUBLE SULPHATES, CHLORIDES AND NITRATES

A-6.0 Outline of the Method

Water-soluble sulphates and chlorides are determined gravimetrically as barium sulphate and silver chloride respectively. Water-soluble nitrates are determined colorimetrically with Nessler's reagent.

A-6.1 Apparatus

A-6.1.1 Distillation Flask

500 ml capacity.

A-6.1.2 Mechanical Shaker

A-6.1.3 Nessler Tubes

50-ml capacity.

A-6.2 Reagents

A-6.2.1 Hydrochloric Acid

Approximately 5 N.

A-6.2.2 Ethyl Alcohol

95 percent (v/v) or rectified spirit (see IS 323).

A-6.2.3 Barium Chloride Solution

10 percent, obtained by dissolving 12.0 g barium chloride crystals in water and making up to 100 ml.

A-6.2.4 Concentrated Nitric Acid

(see IS 264).

A-6.2.5 Silver Nitrate Solution

Approximately 5 percent (m/v).

A-6.2.6 Devarda's Alloy

Containing 45 parts of aluminium, 50 parts of copper and 5 parts of zinc. Heat the aluminium in a crucible in a furnace until it begins to melt. Add copper in small portions and heat until liquefied. Now plunge zinc into the molten mass. Cover the crucible, heat the mixture for a few minutes and stir with an iron rod. Allow it to cool slowly with the cover on and then pulverize the crystalized mass.

A-6.2.7 Sodium Hydroxide Solution

Approximately 20 percent (m/v).

A-6.2.8 Nessler's Reagent

Dissolve 10 g of potassium iodide in 10 ml of water and add to it slowly, with stirring, a saturated aqueous solution of mercuric chloride (HgCl₂) until a slight permanent precipitate forms. Add 30 g of potassium hydroxide, and when it has dissolved, add 1 ml more of mercuric chloride and then dilute to 200 ml with water. Allow to settle overnight and then decant the clear solution. Keep the solution in a bottle closed with well-fitting rubber stopper.

A-6.2.9 Standard Ammonium Chloride Solution

Take 0.017 g of ammonium chloride, dissolve in water and dilute in a graduated flask to 1 000 ml.

A-6.3 Procedure

A-6.3.1 Preparation of Solution

Shake 20 g of the material with 200 ml of water in a chemically resistant glass flask for 1 hour on a mechanical shaker. Filter the mixture and reserve the filtrate for determinations given in A-6.3.2 to A-6.3.4.

A-6.3.2 Determination of Sulphates

Acidify 50 ml of the clear aqueous extract with 3 ml of hydrochloric acid and add a few millilitres of ethyl alcohol. Warm the solution to reduce the chromate which is noted by a change in colour to green. Boil the solution vigorously to drive off organic compounds, care being taken that the solution does not splash. Add excess of barium chloride solution and allow to stand overnight. Filter the precipitated barium sulphate through a Gooch crucible. Wash the precipitate with hot water until washings are free of chloride ions. Dry the crucible containing the precipitate and ignite it over a burner or in the muffle furnace at 600 to 700°C for 30 min. Cool in a desiccator and weigh.

A-6.3.2.1 Calculation

Water-soluble sulphate (as SO_4), percent by mass = 8.23 M

where M is mass in g of barium sulphate precipitate.

A-6.3.3 Determination of Chlorides

Acidify 50 ml of the clear aqueous extract obtained as in A-6.3.1 with 10 ml of concentrated nitric acid and add a slight excess of silver nitrate solution. Coagulate the precipitated silver chloride by warming. Protect the precipitate from light by wrapping with black paper. Filter the precipitate through a sintered glass or Gooch crucible. Wash the precipitate with water containing traces of nitric acid and dry at $100 \pm 5^{\circ}$ C to constant mass.

A-6.3.3.1 Calculation

Water-soluble chlorides

(as Cl), percent by mass

A-7.1.1 Mechanical Shaker

A-7.2 Reagents

A-7.1 Apparatus

A-7.2.1 Dilute Sulphuric Acid

Dilute 25 ml of sulphuric acid (see IS 266) by adding to water, with cooling, and making up to 100 ml with water.

A-7.2.2 Standard Ferrous Ammonium Sulphate Solution

0.1 N (see IS 2316).

A-7.2.3 Standard Potassium Permanganate Solution

0.1 N (see IS 2316).

A-7.3 Procedure

Shake 30 g of the sample with 100 ml of water for 1 h on a mechanical shaker at 27 ± 2°C. Filter and transfer 50 ml of the clear filtrate to a conical flask and acidify with sulphuric acid. Add a measured excess volume (usually 50 ml is sufficient) of the standard ferrous ammonium sulphate solution and titrate the excess against standard potassium permanganate solution.

A-7.4 Calculation

Water-soluble chromates (as CrO₃), percent by mass = $0.022 23 (V_1 - V_2)$ where

 V_1 = volume in ml of standard ferrous ammonium sulphate solution added, and

 V_2 = volume in ml of standard potassium permanganate solution required for titration.

= 4.96 M

where M is mass in g of silver chloride precipitate.

A-6.3.4 Determination of Nitrates

Place 50 ml of the clear aqueous extract obtained as in A-6.3.1 in a distillation flask and dilute to 150 ml with water. Add 3.0 g of powdered Devarda's alloy and 30 ml of sodium hydroxide solution and close the apparatus at once. Warm the flask gently until the reaction starts and allow the reaction to proceed gently for about 30 min. Distil the mixture, collecting the distillate in a receiver containing 30 ml of water and 2 ml of concentrated hydrochloric acid, until about 70 ml of water has distilled over. Keep the receiver cool with running water. Dilute the distillate to 250 ml. Transfer 5 ml of this solution to a Nessler tube and to this add 1 ml of Nessler's reagent. Match the colour produced with a series of similar Nessler tubes to which varying measured quantities of standard ammonium chloride solution and 1 ml of Nessler's reagent have been added.

A-6.3.4.1 Calculation

Water-soluble (as NO3),

percent by mass

= 0.02 V

where V is volume in ml of standard ammonium chloride solution required to match the colour.

A-7 DETERMINATION OF WATER-SOLUBLE CHROMATES

A-7.0 Outline of the Method

Water-soluble chromates are determined volumetrically by adding an excess of ferrous ammonium sulphate to the water extract of the material and then titrating the excess against standard potassium permanganate solution.

ANNEX B

(Foreword)

COMMITTEE COMPOSITION

Raw Materials for Paint Industry Sectional Committee, CHD 021

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Coates of India Ltd, Calcutta

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The Development Commissioner (SSI), New Delhi

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Export Inspection Council of India, New Delhi

Deve Paints Ltd, Mumbai

Goodlass Nerolac Paints Ltd, Mumbai

ICI India Ltd, Calcutta

Indian Paints Association, Mumbai

The Kerala Minerals and Metals Ltd, Quilon

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Ministry of Defence (R&D), New Delhi

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National Organic Chemical Industries Ltd, Mumbai

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Shriram Institute for Industrial Research, Delhi

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Director General, BIS (Ex-officio Member)

Member Secretary

SHRI S. MAZUMDER Joint Director (Chem), BIS

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Pigments and Extenders Subcommittee, CHD 021:01

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Review of Indian Standards

Amendments are issued to standards as the need arises on the basis of comments. Standards are also reviewed periodically; a standard along with amendments is reaffirmed when such review indicates that no changes are needed; if the review indicates that changes are needed, it is taken up for revision. Users of Indian Standards should ascertain that they are in possession of the latest amendments or edition by referring to the latest issue of 'BIS Handbook' and 'Standards: Monthly Additions'.

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Amendments Issued Since Publication

Amend No.	Date of Issue	Text Affected
		A.

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CALCUTTA 700 054 337 86 26, 337 91 20

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60 38 43 \\
60 20 25
\end{cases}$

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